**An Experimental Approach to Understanding Sublimation Water Ice Losses from Planetary Regolith Analogue Mixtures for ESA's PROSPECT Package.** J. I. Mortimer[1], S. J. Barber[1], and M. R. Leese[1], [1]School of Physical Sciences, The Open University, Walton Hall, Milton Keynes, Buckinghamshire, United Kingdom, MK7 6AA, UK. (James.Mortimer@open.ac.uk).

Introduction: ESA's PROSPECT package, which is due to fly to the south polar region of the Moon on board Russia's Luna27 mission in 2024, consists of two main elements: the ProSEED sample drill, and the ProSPA volatile analysis instrument [1]. After drilling down to a depth of up to 1.2 metres, the ProSEED drill will then collect icy regolith material and bring it up to the lunar surface, before transferring it to the ProSPA oven carousel. From there, it will be rotated and positioned under a sample imaging system (SamCam) to work out the volume of the sample material in the oven, then rotated again to a tapping station, where it will be sealed, ready for heating and thus volatile release for analysis in the ProSPA instrument mass spectrometers. However, these processes of sample retrieval, positioning, transfer, imaging, and sealing all take time, during which the icy regolith sample is exposed to radiative and conductive heat sources, thereby potentially raising the temperature of the icy sample. Exposed to the high vacuum conditions of the lunar surface environment, such temperature rises may result in ice mass loss caused by sublimation, and this is important to quantify with some confidence in advance of lunar surface operations.

Previous studies conducted in support of PROSPECT have indicated that significant isotopic fractionation of the remaining water ice is unlikely until more than approximately 30% of the starting water ice mass has been lost [2]. Despite this, it is still necessary to have as much ice content remaining for analysis as is reasonably possible; the current ESA requirement is a maximum loss of 5% of the starting water ice mass across all of the processes outlined above, from retrieval to sealing. This limit on the amount of permissible mass loss then provides constraints on the maximum temperatures of different parts of the PROSPECT systems and so is of high importance to the thermal design of the entire package. Computer numerical simulation can model the expected water ice mass losses at various stages of this sample handling pathway, but these need to be verified by an experimental approach. If the models can be reconciled with real-world data at temperatures and pressures achievable in a laboratory setting, it increases confidence in the ability of the models to accurately predict likely sublimation losses at lunar-relevant temperatures and pressures.

Experimental Procedure: In brief, the set-up comprises a large vacuum chamber with a side-mounted hinged quick access door, on top of which sits a microbalance (CI Precision Recording Microbalance). The microbalance is capable of taking loads up to 5 g and of measuring differences in mass between the counterbalance and the sample of up to a maximum of 500 mg to an accuracy of 0.1 μg. The microbalance is controlled *via* its associated electronics and display box, or through its computer software, through which mass and temperature data (from a K-type thermocouple) are recorded at chosen time intervals (up to 60 measurements per minute) for any user-set experimental duration. Hanging down into the chamber from the microbalance head are two fine NiCr wires, one of which supports the counterbalance, and the other the sample weighing pan. The aluminium foil sample pan is suspended mid-way into a temperature-controlled copper jacket (cooled by liquid nitrogen and heated by a PID-controlled waterproof heating wire circuit), and is cooled from above by a liquid nitrogen cooled copper canopy. Both the jacket and the canopy are thermally isolated from the room-temperature metal walls and floor of the chamber as much as practical by the use of PTFE stilt legs.
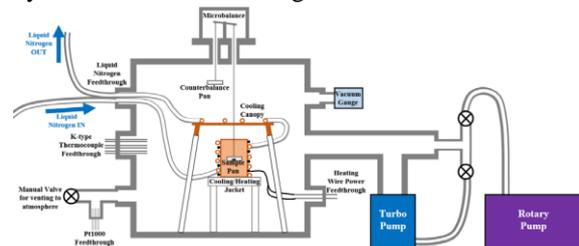
Figure 1: Schematic Diagram of the Sublimation Chamber and Microbalance Set-up

In a typical experiment, regolith simulant is first weighed out directly into the suspended sample pan. Once the sample pan contains a suitable mass of regolith simulant, the chamber is closed and pumped down to low vacuum overnight at room temperature. This removes most of the moisture already absorbed by and adsorbed onto the simulant particles. Immediately before starting an experiment, the chamber is quickly vented to atmosphere. At this point, the microbalance software is set to record both sample pan mass and temperature (typically in the region immediately above the sample pan), so that a record is kept of both the dry simulant starting mass and also the exact mass of water added to the sample pan. A syringe is used to add deionized water to the simulant in the sample pan, drop by drop. The increase in mass on the sample pan is monitored in real-time and recorded by the microbalance software. When the desired mass of water has been added to the sample pan, the pan is removed from the microbalance suspension wire and the regolith and water it contains are quickly mixed with a stainless steel spatula to

ensure a homogenous regolith/water mixture. The sample pan is then re-suspended from the microbalance inside the chamber, thermocouple and resistance thermometer positioning is checked, and the chamber closed again. A manual valve is opened to permit the flow of liquid nitrogen into the copper jacket and cooling canopy inside the chamber. When the temperature in the region of the sample pan has been stable at a minimum temperature (typically around -181 °C), for 5-10 minutes, the chamber is evacuated *via* a rotary vane pump and a turbomolecular pump; the measured base pressure in the chamber is typically around 10^{-6} mbar. As the pressure falls, the temperature of the icy regolith sample mixture on the sample pan begins to rise from near-liquid-nitrogen temperatures as radiative heat transfer through the vacuum overtakes conductive and convective heat transfers in an atmosphere, with the cooling effect of the liquid nitrogen pipework being increasingly offset by the warming effect of the room temperature chamber walls as the pressure falls. Sample temperatures eventually stabilise out (in the experiments conducted to date, sample temperatures range between -40 °C and -95 °C; see Results section; the range is caused by incremental changes to the thermal setup of the chamber in order to achieve lower temperatures).

Initial Results: Water ice losses as percentages of the starting ice mass for various initial experiments conducted with both JSC-1A (Fig.2) and NU-LHT-2M (Fig.3) regolith simulants are presented in this abstract. A range of water ice contents and temperatures of sublimation have been investigated, across timescales ranging from approximately 1 hour to over 4 hours.
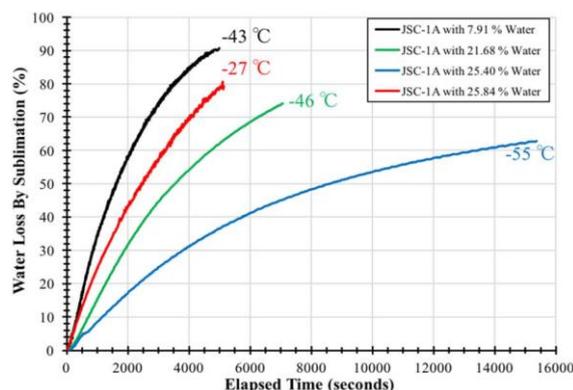

Figure 2: Water Ice Mass Loss Percentages over Time for Icy Regolith Mixtures made using JSC-1A simulant

In each case, the rate of water ice mass loss is observed to reduce as time passes, starting with a relatively rapid mass loss and becoming slower as progressively more water ice is lost. This is attributed to an increasing thickness of dehydrated insulating regolith simulant overlying the sublimation front at the top surface of the shrinking water ice mass as sublimation progresses, insulating the remaining ice

and/or providing a more tortuous pathway for water vapour molecules to navigate before being lost from the sample. In comparison, experiments with water ice only (i.e. no regolith) display perfectly linear water ice mass loss rates over time.
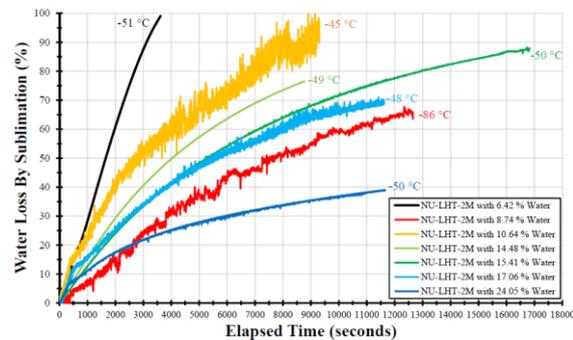

Figure 3: Water Ice Mass Loss Percentages over Time for Icy Regolith Mixtures made using NU-LHT-2M simulant

Interim Conclusions: All icy regolith samples, whether made with JSC-1A or with NU-LHT-2M, display logarithmic mass loss trends, in contrast to the linear mass loss behaviour of pure water. The more rapid initial rate of mass loss observed in icy regolith samples is comparable to that of the rates of pure water ice loss measured in this same study, for samples of similar exposed surface areas and temperatures. However, as sublimation progresses and the sublimating ice front moves deeper down into the sample mass, the rate of mass loss decreases. Given the importance of assumptions about sample surface areas to rates of mass loss when comparing experimental with modelled data, future experiments will use a fully representative part of a real ProSPA (prototype) oven with the microbalance, in order to more accurately replicate the sample geometries of the mission scenario. All samples at temperatures investigated so far (down to -86 °C) lose a mission-relevant 5 % water ice mass within the first 680 seconds or less, with warmer samples losing 5 % much more rapidly than colder samples (Table 1). The addition of multi-layer insulation to the chamber walls will aid the investigation of mass loss rates at lower temperatures, down to -100 °C.

Regolith Simulant	Initial Water Ice %	Initial Water Ice Mass (mg)	Temperature (°C)	Time taken to lose X % (seconds)		
				1%	2%	5%
JSC-1A	7.91	20.1857	-43	50	100	200
JSC-1A	21.68	65.0325	-46	150	230	430
JSC-1A	25.4	80.2928	-55	220	300	560
JSC-1A	25.84	81.8704	-27	50	90	110
NU-LHT-2M	6.42	18.1719	-51	120	160	260
NU-LHT-2M	8.74	34.4581	-86	100	300	680
NU-LHT-2M	10.64	24.8893	-45	50	70	150
NU-LHT-2M	14.48	34.1953	-49	50	70	240
NU-LHT-2M	15.41	36.6258	-50	100	150	400
NU-LHT-2M	17.06	41.7635	-48	80	100	250
NU-LHT-2M	24.05	64.5955	-50	50	100	260

Table 1: Time taken to lose 1%, 2% and 5% water ice mass

References: [1] Trautner R. et al. (2018) 69th International Astronautical Congress, Paper ID: 42773 [2] Mortimer J. et al. (2018) *Planet. Space Sci., 158,* 25-33.